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PART A. HYDROCARBON SUSPENSION IN SLUSH HYDROGEN

PART B. HEAT TRANSFER TO SLUSH HYDROGEN

U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS REPORT

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PART A.   HYDROCARBON SUSPENSION IN SLUSH HYDROGEN   ✓

PART B.   HEAT TRANSFER TO SLUSH HYDROGEN   ✓

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◆NBS◆

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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ABSTRACT

Methods of preparing suspensions of a hydrocarbon
in slush hydrogen were investigated. Suspensions were
prepared using approximately 5000 ppm by mass of methane,
ethane, or cyclopropane in slush hydrogen. The suspensions
were stable in the slush, but the hydrocarbons settled out of
the liquid melt.

Heat transfer to slush hydrogen was measured at
one atmosphere and at triple-point pressure. The data
were compared with those for heat transfer to liquid hydro-
gen, and to classical heat transfer correlations for nucleate
boiling. The slush data fit convective heat transfer correla-
tions quite well. In general, the data show that for a given
heat flux, the temperature difference between the wall and
the bulk liquid is not as highly influenced by pressure as
predicted by the correlation for nucleate boiling.

Key Words: Boiling heat transfer; convective heat transfer;
heat transfer; hydrocarbon in liquid hydrogen; hydrocarbon
suspensions; liquid hydrogen; slush hydrogen.

PART A. HYDROCARBON SUSPENSION IN SLUSH HYDROGEN

PART B. HEAT TRANSFER TO SLUSH HYDROGEN

Charles F. Sindt

Introduction

The Cryogenics Division of the National Bureau of Standards has been involved in a study of the characterization of slush hydrogen for several years. This analytical and experimental program has been directed toward investigation of the characteristics that apply to the use of slush hydrogen as a propellant for rockets. Hydrogen has been chosen for a variety of chemical and nuclear rocket vehicle applications because of its low molecular weight, high heat capacity, and high specific heat ratio. Subcooled liquid hydrogen, slush hydrogen, and solid hydrogen have been under consideration as propellants for some time, because they can extend the duration of space missions, especially deep space missions. The extent of these advantages is predictable for specific mission requirements and is dependent upon the ability to use increased heat capacity and increased density to augment thrust-to-weight ratios and extend mission duration.

To apply the benefits of slush as a propellant, characteristics of preparation and management must be explored and developed. Characteristics previously investigated include those of preparation, flow, pumping, aging, solid particle configuration, and instrumentation. Characteristics common to most applications, which have not been investigated or have been studied very little, are those of mixing and heat transfer.

Nuclear rocket engines have a unique requirement for carbon in the hydrogen propellant to minimize corrosion of the nuclear fuel elements. Carbon in the form of hydrocarbon suspensions is of interest

in this regard. Prior attempts to prepare hydrocarbon suspensions as colloids in liquid hydrogen have not been successful. The possibility of slush hydrogen enhancing the suspension of a hydrocarbon generated the interest for this study.

Combined with the hydrocarbon suspension investigation was an experimental program designed to measure heat transfer properties of slush and an analytical program to evaluate mixer design and power requirements for mixing slush. The mixing analysis was completed and reported previously in NBS Report 10 717 (McConnell). The heat transfer and hydrocarbon suspension phases of the program are subjects of this report. *

PART A. HYDROCARBON SUSPENSION IN SLUSH HYDROGEN

Charles F. Sindt

1. 0 Introduction

Various attempts have been made to produce a colloid suspension
of a hydrocarbon in normal boiling liquid hydrogen, and one experiment
was conducted using slush hydrogen [Vander Wall, 1970, and Bartlit, 1970].
These attempts were unsuccessful in development of a colloid; however,
suspensions resulted in several cases, but the hydrocarbon content was
much higher than the 5000 ppm considered necessary for fuel element
corrosion inhibition. Excess hydrocarbon in the hydrogen propellant
results in a serious degradation of the rocket engine's specific impulse
since specific impulse is nearly proportional to the square root of molecu-
lar weight of the propellant. Therefore, it is important to select hydro-
carbons of low molecular weights and not to exceed 5000 ppm. The most
desirable hydrocarbon is methane, then probably ethane, butane, and
propane, in that order. This experimental program was conducted using
methane, ethane, and cyclopropane as these were the most successful
candidates used in previous work [Vander Wall, 1970].

2. 0 Objectives

The program was divided into two series of tests, both with the
same overall objective of producing a stable suspension of approximately
5000 ppm by mass of hydrocarbon in slush hydrogen. Another objective
was to maintain the hydrocarbon in a stable suspension even after melting
of the solid hydrogen in the slush.

In the first series of tests, the hydrocarbon was injected into the
liquid hydrogen prior to slush preparation. The hydrocarbons were
mixed with hydrogen gas; the methane and cyclopropane mixtures were
10 mole percent and the ethane was 3 mole percent in hydrogen. These

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concentrations were selected because Vander Wall [1970] reported they gave the best results for forming suspensions in liquid hydrogen. In the second series of tests, the hydrocarbon was injected into the slush hydrogen _after_ slush preparation. The hydrocarbons used for injection in this latter series were pure gases.

3.0 Experimental Apparatus

A 10-cm diameter vacuum-insulated glass vessel with a usable volume of 2000 cm^3 was used for the hydrocarbon suspension investigation. This vessel was completely submerged in a liquid hydrogen bath which could be maintained at any desired temperature between hydrogen triple point and normal-boiling point temperatures. The bath dewar was closed and the temperature of the hydrogen was adjusted by controlling the pressure over the liquid. This dewar was suspended in a liquid nitrogen filled dewar for thermal radiation protection of the liquid hydrogen bath. All of the vessels were made of glass to permit visual and photographic observation. A schematic of the system is presented in figure 1, and a photograph of the experimental vessel is shown in figure 2.

The two hydrogen containing vessels could be pumped independently by two separate vacuum pumps or they could be manifolded together to one pump. The experimental vessel was connected to a 0.0071 m^3/s pump which discharged through a dry gas flow meter. Vessel pressure was maintained by a barostat capable of controlling set pressure to $\pm$ 135 N/m^2. The bath dewar was connected to a 0.052 m^3/s pump and was maintained at the desired pressure with either the barostat or a manostat. This arrangement permitted control of the experimental dewar pressure separate from the bath dewar and provided a means of measuring the volume of hydrogen gas pumped to produce slush in the

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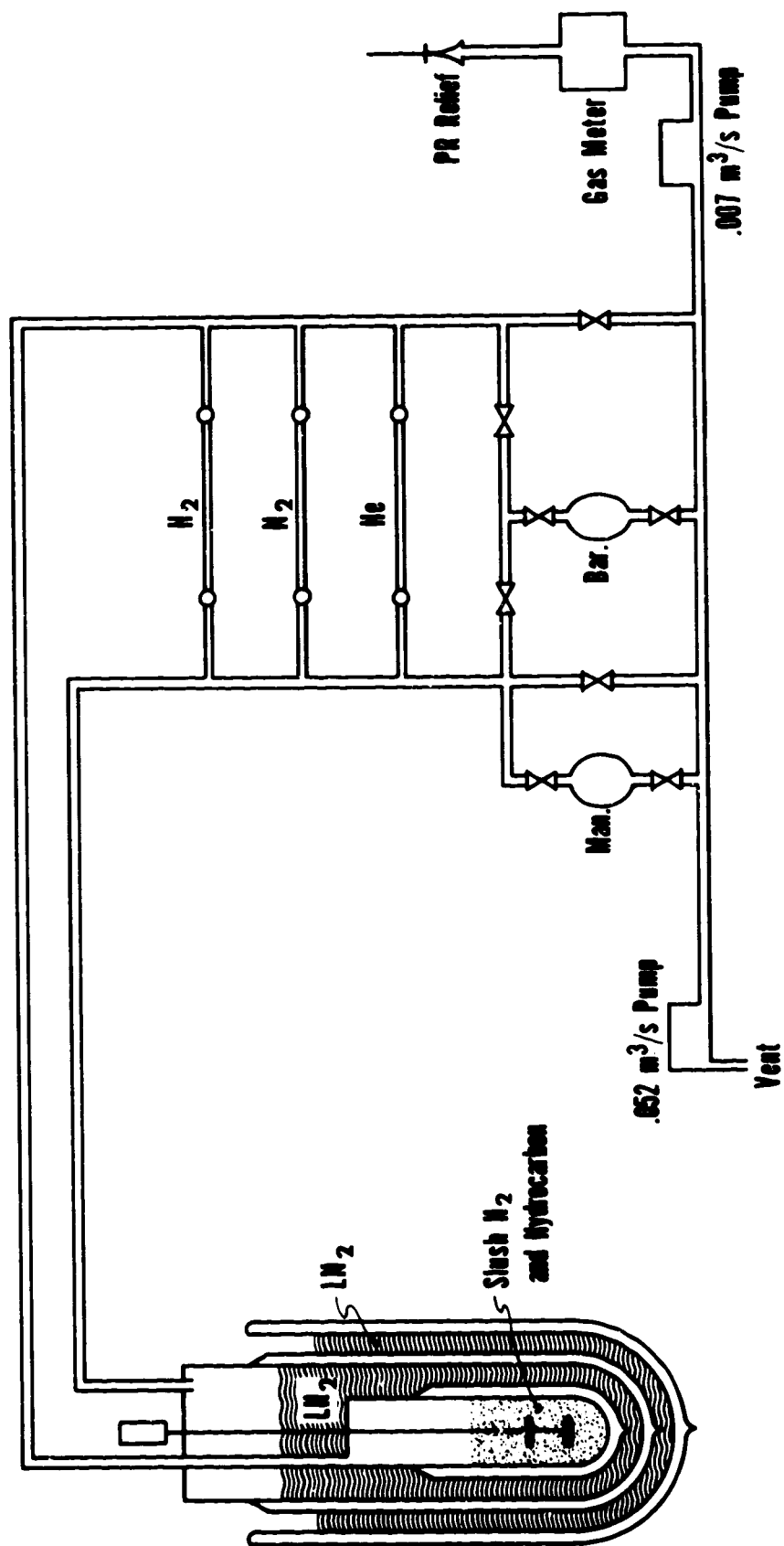
Figure 1.   Hydrocarbon Suspension System Schematic

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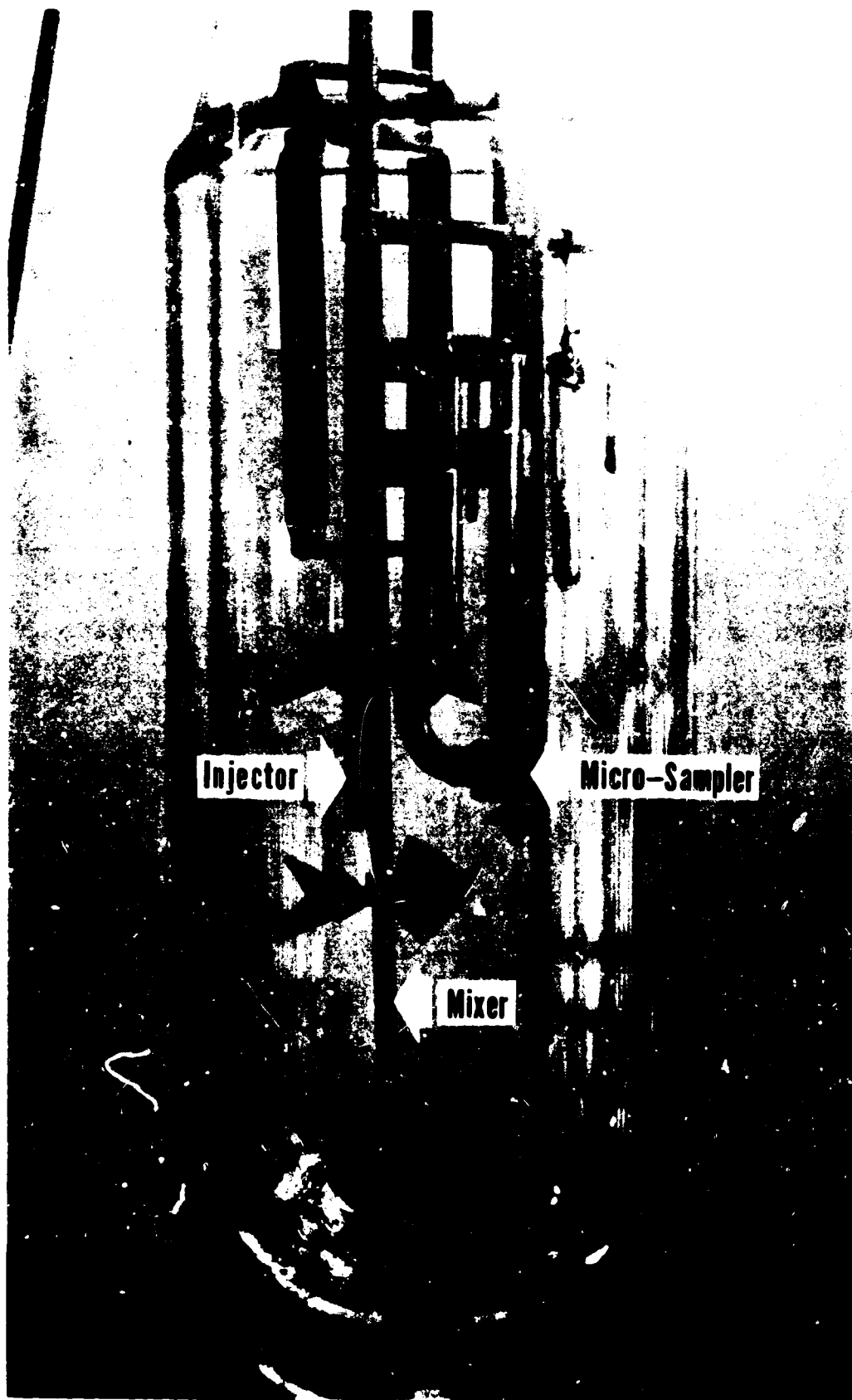
Figure 2.   Experimental Dewar

experimental vessel. Both dewars were independently pressurized with hydrogen, helium, or nitrogen gas for purging and for pressurization.

Within the experimental dewar was a mixer which was used for slush preparation and for homogeneous mixing of the hydrocarbons during injection and during sampling of the mixture.

The hydrocarbon injection probe, shown in figure 2, was vacuum insulated and was heated with helium gas to prevent condensation and freezing of the hydrocarbon prior to introducing it into the liquid hydrogen. The probe had a 0.33-mm-diameter injector at the bottom and could be raised or lowered to the desired injection level.

3.1 Hydrocarbon Sampler

The hydrocarbon content was determined by collecting a small sample of the mixture and analyzing it in a hydrocarbon analyzer. The sample was collected in a micro-sampler of approximately 1 cm^3 volume. This sampler was mounted on a probe that extended from the top plate into the experimental dewar. The sampler could be lowered into the mixture near the dewar bottom and raised into the ullage above the liquid.

A small tube connected the sample volume to a sample collector with two chambers. The tube and the sampler were both enclosed in heaters which were used to vaporize the hydrogen and hydrocarbon. The gases were collected in the chambers and were later analyzed with a hydrocarbon analyzer using a hydrogen flame-ionization detector capable of determining 10 ppm by mass of carbon. The sample probe can be seen in figure 2 and a schematic of the sampling system is shown in figure 3.

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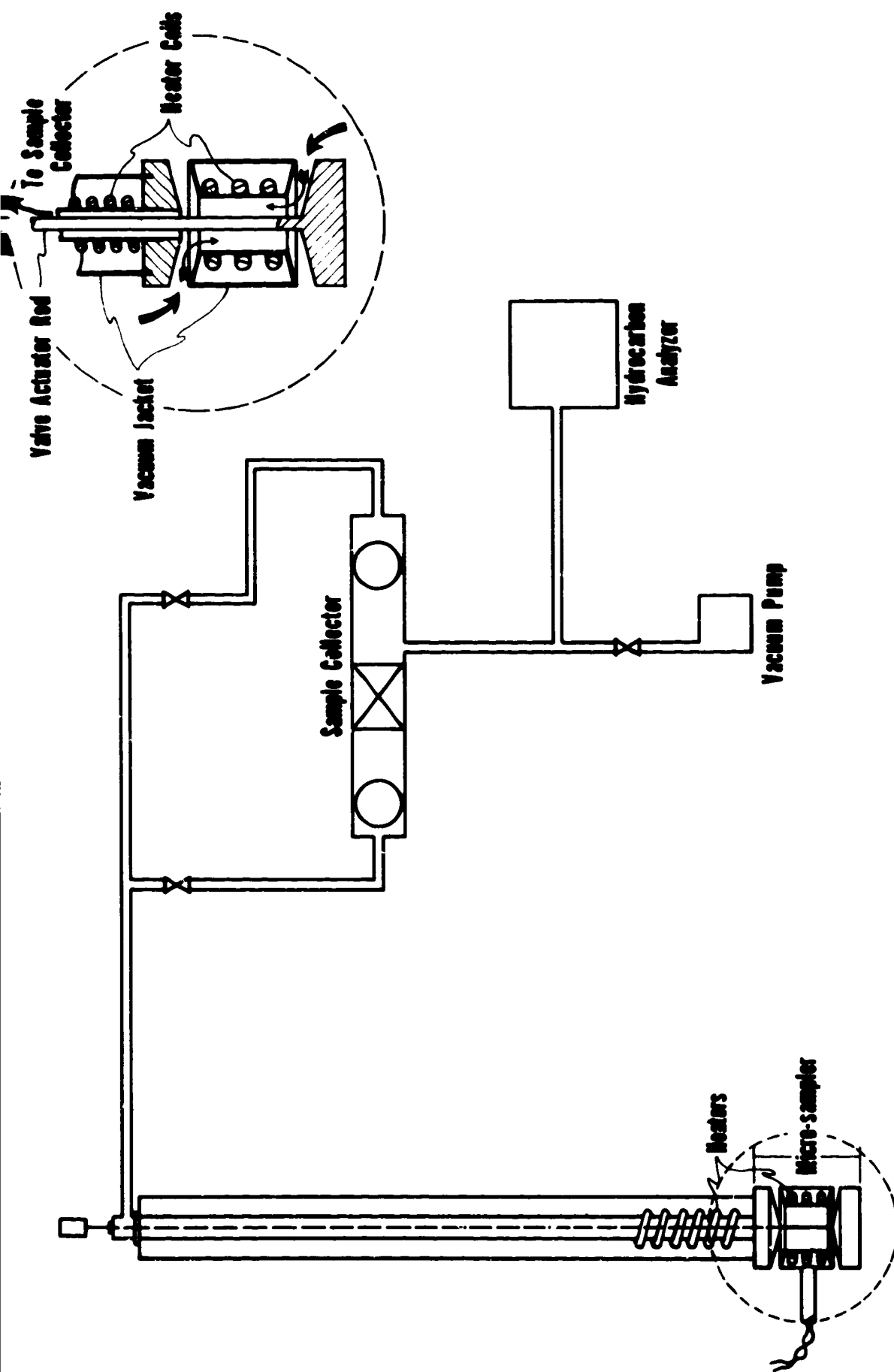
Figure 3. Hydrocarbon Sampler Schematic

4.0 Experimental Procedure

4.1 Injection of Hydrocarbons Prior to Slush Preparation

In the experiments where the hydrocarbon was added to liquid hydrogen prior to slush preparation, the liquid hydrogen was first cooled to triple-point temperature by pumping the bath and the experimental vessel to triple-point pressure. The mixture of hydrocarbon and hydrogen gas was then injected into the liquid hydrogen 6 to 12 cm below the liquid surface. The injection gas pressure was held at approximately 380 kN/m^2 above the dewar pressure. During injection, the liquid was vigorously mixed to prevent accumulation of hydrocarbon around the end of the injection probe. Timing the period of injection proved to be a fairly consistent means of reproducing previous hydrocarbon concentration levels.

After injection, the hydrocarbon mixture was again cooled to triple-point temperature by pumping the hydrogen bath. At this time the liquid volume in the vessel was determined from liquid level measurement, and samples were taken for hydrocarbon content. After sampling, slush was prepared using the "freeze thaw" technique described by Mann, et al. [1966].

Immediately after slush preparation, the volume of the mixture was again measured. The general texture of the slush mixture was observed visually and photographically while it was allowed to age in the experimental vessel. After the desired aging period was complete and observations were made, the solid hydrogen was melted by adding heat with the sampler heater. The liquid volume was again measured while the liquid hydrogen-hydrocarbon mixture was at hydrogen triple-point pressure and temperature. From the volume of slush and the volume of liquid at triple point, the slush solid fraction was calculated.

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After the liquid volume was determined, the mixture was again sampled to determine hydrocarbon content. These samples were compared to samples taken prior to slush preparation with suitable corrections made for the mass of hydrogen removed in making slush.

4.2 Injection of Hydrocarbons After Slush Preparation

In the experiment where hydrocarbons were added after slush had been prepared, the general procedure was the same except that slush was prepared before injection and the injection gas was pure hydrocarbon. Also the slush degradation due to injection was determined by volume measurements made prior to and after injection.

Slush solid fraction in both test series was generally between 0.45 and 0.5 immediately after preparation. When hydrocarbon injection was accomplished after slush was formed, the solid fraction was degraded because of the heat added by the hydrocarbon.

4.3 Hydrocarbon Sampling Procedure

Sampling of the hydrogen-hydrocarbon mixture proved to be difficult as consistent samples were not easily obtained. In an effort to get as much reproducibility as practical, a sampling procedure was established that gave reasonably consistent values in the liquid hydrogen-hydrocarbon mixture but was not acceptable for sampling of slush.

The sampling procedure started by evacuating both sample collection chambers with the vacuum pump. The dewar contents were vigorously stirred with the mixer. While stirring the mixture, the sampler was lowered into the vessel and both ends were opened an equal amount to allow the mixture to flow through the sampler. Stirring continued until the sampler was closed and was raised into the ullage above the mixture. The valve to the first collection chamber was opened and heat applied to the sampler and to the tube connecting the

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sampler with the collection chamber. When the sampler was above the liquid hydrogen boiling temperature, the first collection chamber valve was closed and the valve was opened to the second chamber. More heat was applied until the temperature in the sampler was well above the boiling temperature of the hydrocarbon in use. The valve to the sampler was closed and the valve between chambers opened. The contents of the two chambers were then mixed together after which they were analyzed with the hydrocarbon analyzer.

Since the hydrocarbon melted and vaporized after the hydrogen, and since the amount of hydrocarbon was small, most of the hydro-carbon would have been left in the sampler if a single chamber had been used for collection. The use of the second evacuated chamber caused the vaporized hydrocarbon to expand as it warmed. Because the volume of the second chamber was 300 times larger than the sampler volume, the remaining hydrogen gas and the hydrocarbon in the sampler were recovered; therefore, well over 99 percent of the hydrocarbon was collected in the second chamber. The gases in the chambers were mixed with steel balls that were caused to roll back and forth within the collection chambers. This sampling procedure gave the most con-sistent results of the several procedures tried.

Sampling in triple-point liquid hydrogen proved to be most repeatable; however, consecutive samples still varied as much as 30 percent. Capturing a representative sample of the solid hydrocarbon is not a straightforward procedure. Several reasons for the problem could be: 1) the mixture is not homogeneous even though mixing is vigorous, 2) the solid particles of hydrocarbon do not flow through the sampler in a homogeneous manner even though the bulk appears to be nearly homogeneous, and 3) the hydrocarbon solids have an

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affinity for collecting on all surfaces and the washing effects of the agitated fluid hydrogen may not be consistent in the micro-sampler.

Although the problems of sampling were always apparent, averaging as many as 8 consecutive samples and comparing the results with the volume of gas injected gave confidence that the average of the samples was within plus or minus 25 percent of the actual value. Since the desired 5000 ppm was not a precise requirement, no further work was done on improving the sampling technique.

Because actual content of the hydrocarbon in the settled slush, and in the liquid over settled slush, was important in establishing the dispersion of the hydrocarbon, a method was devised to verify the dispersion of the hydrocarbon in slush. The hydrocarbon content in liquid over the settled slush and the average hydrocarbon content of the total mixture (after melting the solid hydrogen) were determined by the method described above. Hydrocarbon distribution in the settled hydrogen solids was verified to be nearly uniform by a special experiment.

The experiment to determine the hydrocarbon dispersion in settled slush consisted of forming the slush hydrocarbon mixture in the usual manner. The solids were then allowed to settle. After the solids had settled, the sampler was lowered into the clear liquid to a point just above the solids and the heater in the sampler was energized. This technique caused warming of the liquid and melting of the settled solid hydrogen from the top. Since the hydrocarbon melting points were all well above liquid hydrogen temperature, the solid hydrocarbon remained and continued to settle on the top of the settled slush remaining in the vessel. The settled hydrocarbon layer could be visually observed because the hydrocarbon particles appear very white and the slush hydrogen particles appear wetted and translucent. The growth of the

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hydrocarbon layer, the measurement of volume change of the liquid, and the total hydrocarbon content were used to determine the amount of hydrocarbon suspended in the settled slush prior to melting. Experimental results of this test are presented in section 5. 3.

5. 0 Experimental Results

5. 1 Injecting Hydrocarbon Prior to Slush Preparation

A series of ten experiments was conducted to determine the effects of adding enough hydrocarbon to produce 5000 ppm by mass of carbon in the hydrogen. In these tests, the hydrocarbon was added to triple-point liquid; slush was then made to a solid fraction between 0. 4 and 0. 55.

The hydrocarbons, methane, ethane, and cyclopropane, were each added in separate experiments. As the hydrocarbons were added through the injector, they formed flocculent solid particles that settled slowly in the triple-point liquid hydrogen. The particles of ethane appeared to be the most flocculent; however, no quantitative data were taken on solid particle size or settling rates. Solid particles from all three of the hydrocarbons settled in the liquid hydrogen so none formed a colloid or a stable suspension.

After injection, slush preparation was accompanied by vigorous mixing of the hydrogen-hydrocarbon mixture. Slush was prepared until solids in the mixture occupied the same volume as the liquid. The resulting mixture did not exhibit jelly characteristics, but was a semi-fluid mixture that could be stirred and had very little tendency to free stand; instead, it flowed quickly to a level surface as a Newtonian fluid. This mixture was, therefore, intermediate to a settled slurry and a true gel, since a gel is actually a colloid and a slurry settles to a densely packed bed.

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The addition of 5000 ppm by mass of the hydrocarbons did not grossly affect the solid fraction at which the solid hydrogen in slush fills the liquid volume. In nearly all of the experiments, the addition of 4000 to 5000 ppm hydrocarbon reduced by about 0.05 the solid fraction where solid and liquid occupy the same volume. The mixtures were not significantly different, in mixing or settling characteristics, from pure slush hydrogen. A dewar of slush with 6500 ppm methane is shown in figure 4. The slush has aged approximately 15 minutes so some settling (which accompanies aging) has occurred and the resulting clear liquid hydrogen over the settled solid is apparent. The clear liquid was sampled for hydrocarbon content and had less than 20 ppm by mass of hydrocarbon in solution and suspension.

Aging and mixing of the slush and hydrocarbon mixture did significantly affect the character of the solid hydrocarbon particles. After the solid hydrogen in slush had melted, the settled hydrocarbon particles occupied much less volume than they had immediately after injection. A typical change is shown in figures 5 and 6. As is evident in the figures, the volume reduction of settled solid is more than half. This characteristic is important, in that the amount of hydrocarbon required to totally fill the liquid volume, after the hydrocarbon-slush mixture is aged and agitated, is more than twice as large as would be indicated by the flocculent particles that develop at injection.

5.2 Injecting Hydrocarbon After Slush Preparation

Experiments were conducted injecting methane, ethane, and cyclopropane into slush hydrogen at approximately 0.47 solid fraction. The hydrocarbons injected were 99 percent, or higher, purity gases.

In the experiments with methane injection, the solid fraction of the slush was reduced from 0.47 to 0.37 by methane content of 4000 ppm

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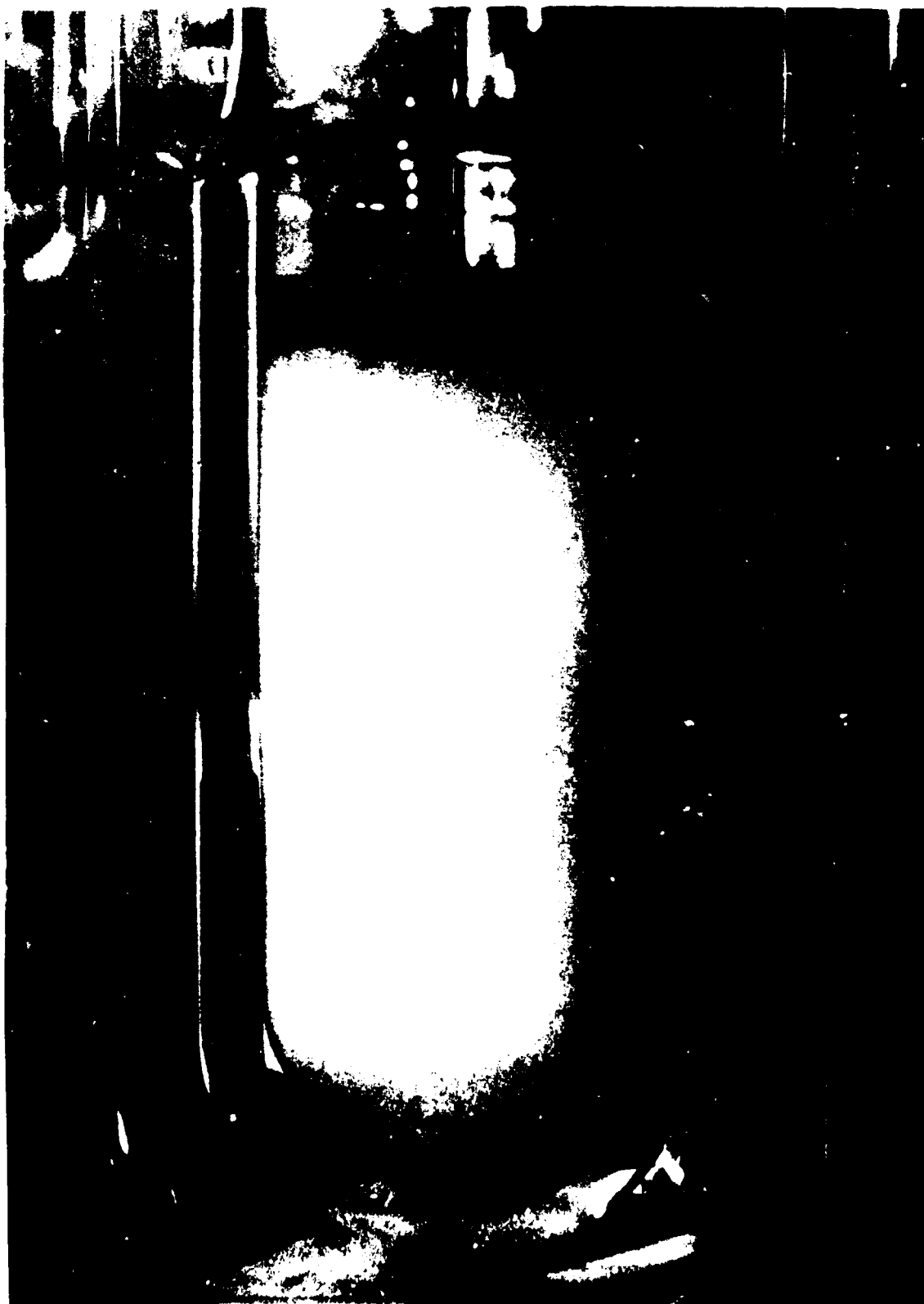
Figure 4. Slush Hydrogen With 6500 ppm Methane

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Figure 5. Cyclopropane Immediately After Injection

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Figure 6.   Cyclopropane Five Hours After Injection

by mass. This reduction in solid fraction during injection occurs because the hydrocarbon gas must be cooled from above its liquefaction point, condensed, further cooled, frozen, and again cooled to the triple-point temperature of hydrogen. The required refrigeration is supplied at the expense of melting the solid hydrogen in the slush. The quantity of solid melted experimentally was in good agreement with the calculated value for the mass of methane injected.

The resulting mixture definitely was not homogeneous, as clear liquid hydrogen was visible over the settled solids. The hydrocarbon appeared well dispersed within the settled solid region but settled out of the liquid very soon after mixing was stopped.

Results of ethane injection were very similar to methane injection. The significant difference was that the slush degradation from injection was from 0.47 to 0.29 solid fraction. In this particular experiment the solid reduction was greater than the calculated value for the 5500 ppm of ethane injected, probably because of the ethane being injected at a temperature well above its liquefaction point. The mixture after injection had considerable clear liquid over the settled solids.

Injection of cyclopropane into slush resulted in melting the solid fraction from 0.47 to 0.15, this solid fraction reduction corresponding to the injection of about 7800 ppm cyclopropane. Since the thermo-dynamic properties of cyclopropane are not available in this temperature region, the calculation for theoretical mass of melted solids was not available.

In all of the experiments, the mixtures resulting after injection displayed no characteristics different than similar mixtures prepared by injection prior to slush preparation. Since the injection always melted a considerable amount of solid hydrogen, this method of adding hydrocarbon was considered less desirable than injection prior to slush preparation.

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5.3 Results of Special Tests

A group of special tests was conducted using ethane as the hydro-carbon, to determine the effect of hydrocarbon concentration on the volume of settled solids in slush. This effect was determined by finding the hydrocarbon content and the slush solid fraction when settled solids occupied the same volume as the liquid; that is, when settled solid level and liquid level were the same.

The experimental procedure was the same as for all of the tests where hydrocarbon was injected prior to slush-making. Enough ethane was injected for a 5000 ppm mixture, then slush was made until the solids and liquid occupied the same volume. Solid fraction of the slush was determined. Then the solid hydrogen was melted and more ethane was added. This cycle was repeated adding more ethane each time until finally the ethane solid completely filled the liquid hydrogen and no solid hydrogen remained. The experimental results are shown in figure 7. Two points to note in figure 7 are: 1) the effect of the hydro-carbon is not linear; and 2) the amount of ethane required for liquid hydrogen is twice that reported by Vander Wall [1970]. This difference is quite probably due to the mechanical agitation during slush prepara-tion and may be due to aging but is the same effect as was shown in figures 5 and 6. The results are important since any propellant system would have turbulence and mechanical agitation in piping, valves, and pumps; and would, therefore, result in similar reduction in volume of the settled/aged hydrocarbon.

A second group of special experiments was conducted to prove that the hydrocarbon was evenly dispersed within the settled solid region. These tests were required because sampling in settled slush was not reliable. The procedure for these experiments was covered in section 4.3. Results were satisfactory, in that the layer of settled hydrocarbon
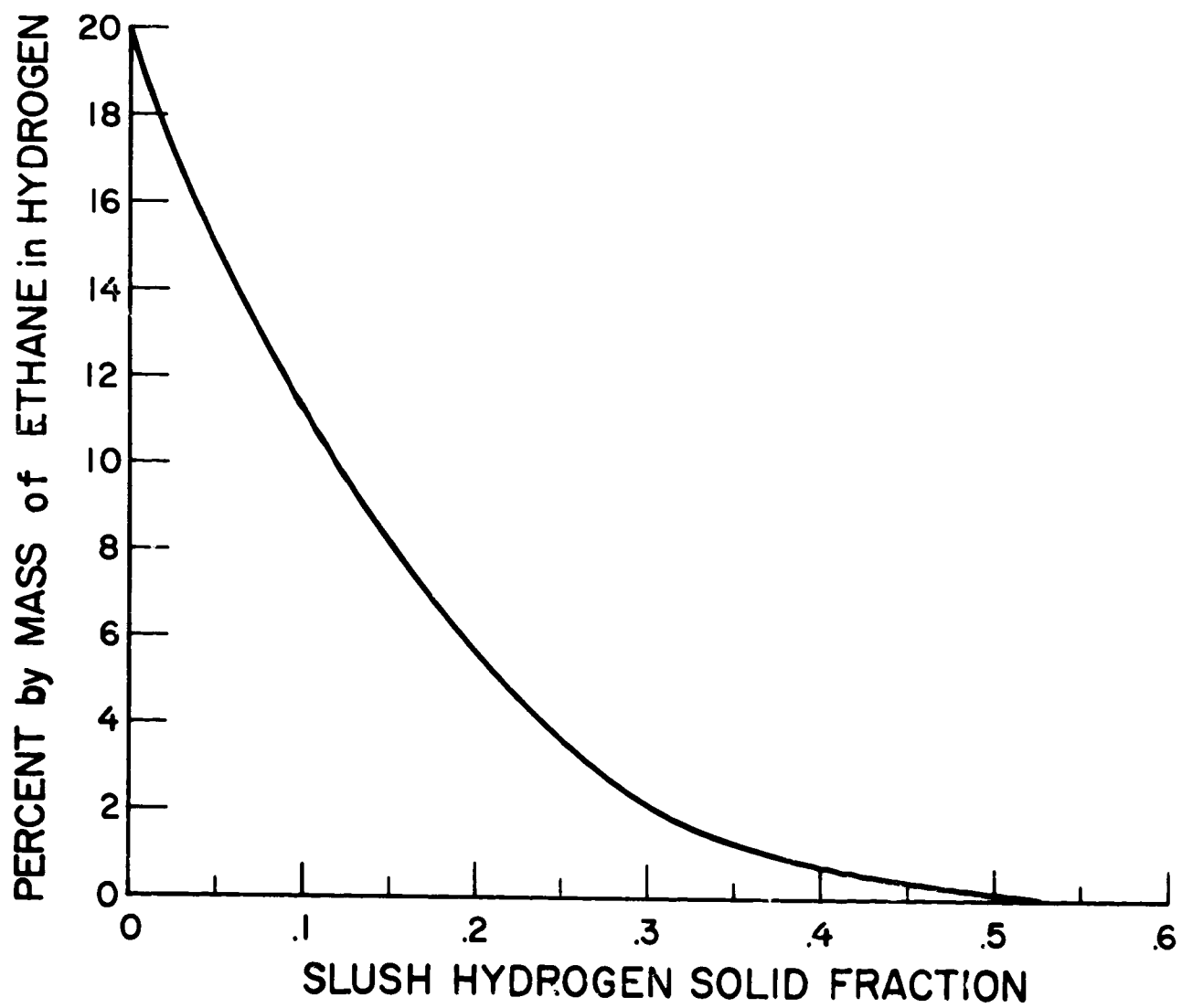
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Figure 7.   Maximum Slush Solid Fraction and Ethane in Liquid Hydrogen

was clearly visible on the settled slush. Photographing the layer proved difficult through the six layers of glass. The results are apparent in a photograph, however, and are shown in figures 8 through 10. The gradual and proportional build-up of the settled solid hydrocarbon layer as the solid hydrogen melted was definite proof that the hydrocarbon was evenly dispersed in the settled slush.

6.0 Conclusions

From the two types of tests conducted in this program, a number of conclusions are evident. Some are positive and others negative with reference to favorably meeting the objectives. The positive conclusions are:

1) A technique for dispersing a hydrocarbon in hydrogen is to prepare slush from the desired mixture of hydrocarbon and triple-point liquid hydrogen. The dispersion is retained in the settled slush hydrogen in a form that is stable as long as significant amounts of solid hydrogen are not melted.

2) Methane is as good as any of the three hydrocarbons tried for preparing the required mixture and is most desirable because of the least degradation of specific impulse of the rocket engine.

3) Preparation of a mixture of liquid hydrogen and solid methane particles is simple in that methane is injected into the liquid through small orifices. The methane is injected as a 10 mole percent mixture in hydrogen gas. Higher mole percent mixtures may work as well.

4) Injection in liquid prior to forming slush is more successful than injection into slush.

5) 5000 ppm by mass of methane in slush hydrogen does not significantly affect the handling characteristics, such as mixing.
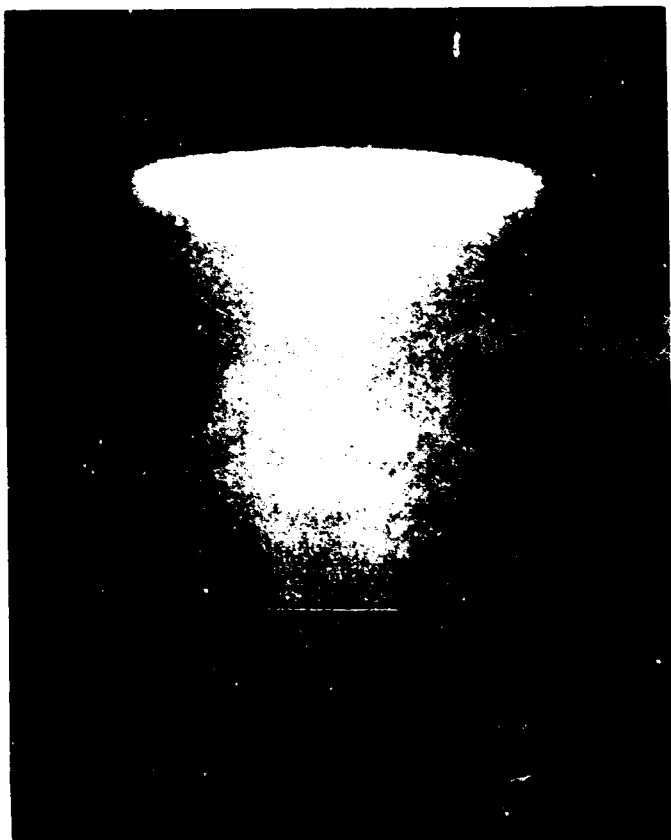
Figure 8.   Layer of Methane on 0.27 Solid Fraction Settled Hydrogen
              Slush-Hydrocarbon Mixture
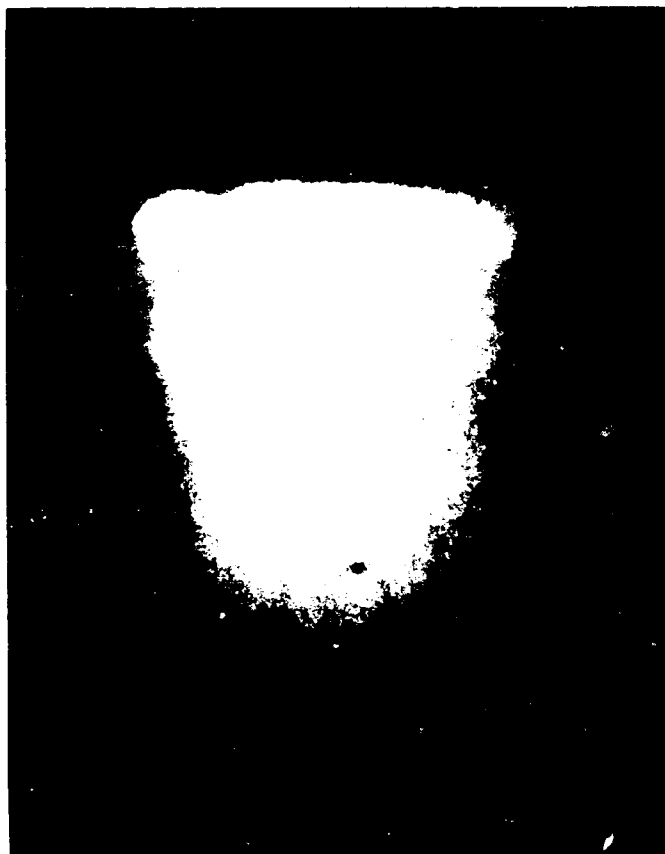
Figure 9.   Layer of Methane on 0.2 Solid Fraction Settled Hydrogen
Slush-Hydrocarbon Mixture

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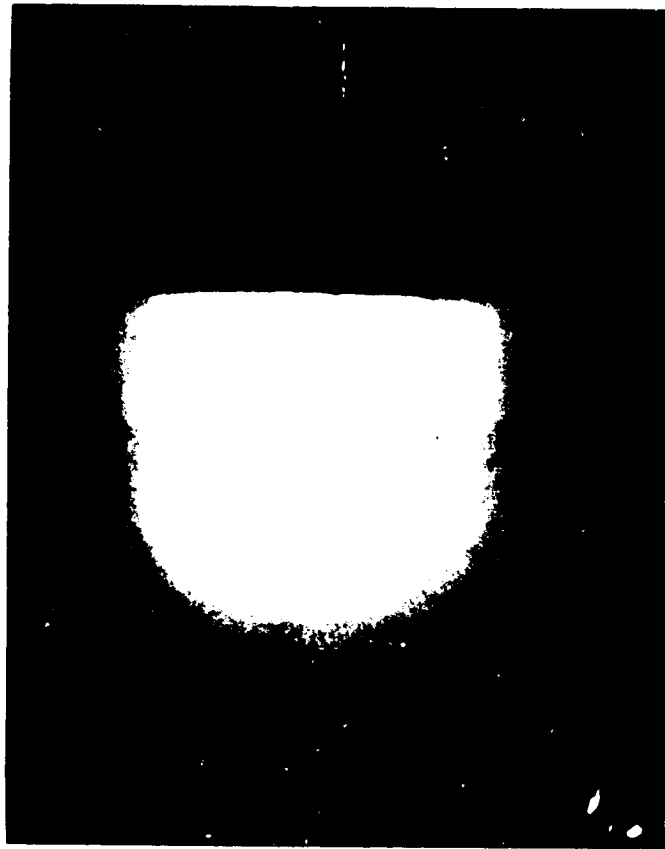
Figure 10. Layer of Methane on 0.11 Solid Fraction Settled Hydrogen
Slush-Hydrocarbon Mixture

The negative conclusions from the experiments are:

1) None of the techniques tried resulted in a dispersion of the hydrocarbon that was stable in liquid hydrogen; therefore, the dispersion only exists in the settled slush portion of the hydrogen.

2) Accurate measurement of the hydrocarbon content in settled slush hydrogen is difficult; consistent representative samples were not obtained nor was a technique developed to obtain a representative sample in settled slush without melting all of the solid hydrogen in the vessel.

3) Mechanical agitation and aging break up the more flocculent particles of hydrocarbon which originally form at injection, and the resulting small particles are still much too large to form a colloid in liquid hydrogen. These particles, therefore, settle out of the liquid very quickly.

4) A significant amount of mixing is required to disperse the hydrocarbon particles in liquid hydrogen. This mixing energy is estimated to be in excess of that required to mix slush hydrogen to a homogeneous mixture.

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PART B. HEAT TRANSFER TO SLUSH HYDROGEN

Charles F. Sindt

1.0 Introduction

Before selecting an apparatus and heat transfer rates to be investigated in the experimental phase of this program, a literature search was made covering the general subjects of storage of cryogenic propellants, e.g., heat transfer to hydrogen, cryogenic propellant stratification, heat transfer to solid-liquid mixtures, and propellant behavior at low and high gravity conditions. After reviewing the information from the literature search, the conclusion was that the initial experimental program should be basic, since no data of value were available on heat transfer to slush and since aerospace applications may require heat transfer information for a large range of heat fluxes. The literature search also revealed several recent papers on heat transfer experiments to normal-boiling-point liquid hydrogen. One such paper on the design and results of an experimental apparatus of laboratory size was published by Coeling and Merte [1968]. This apparatus, and the range of data presented, appeared to be similar to results desired; therefore, a similar guarded heater unit was selected for the slush hydrogen apparatus. This heater design was installed into the system used for the hydrocarbon suspension studies described in section 3.0 of Part A. One distinct advantage of the selection of the heater unit similar to Coeling's was that data were available for direct comparison of heat transfer to liquid hydrogen near normal boiling temperature.

2.0 Objectives

The objectives of this program were: 1) to provide information on heat transfer to slush hydrogen in the range of heat flux that might be expected in aerospace applications, 2) to obtain as much basic

information on heat transfer to slush hydrogen and liquid hydrogen as
time would allow, and 3) to experimentally measure thermal gradients
in a vessel containing liquid or slush hydrogen when the heat flux was
known.

3.0 Experimental Apparatus

The experimental apparatus for heat transfer experiments was the
same as that used for the hydrocarbon suspension work described in
Part A. The only differences were that the heat transfer unit was installed
in the 10-cm-diameter vessel and the injection probe and micro-sampler
probe were removed. The mixer was retained in the same configuration.

3.1 Heat Transfer Unit

The heat transfer unit consisted of a 2.54 cm diameter cylindrical
block of electrolytic tough pitch copper. This cylinder was 1.9 cm long
and was drilled in six equally spaced places to accept six carbon resistors
which were used as the heat source. The carbon resistors were embedded
in a high thermal conductivity epoxy and were connected in parallel con-
figuration. The electrical resistance of the unit was 13.86 ohms at 20 K.
A schematic of the heater unit is shown in figure 11.

The surface of the heater exposed to the hydrogen was formed by
a 0.05 mm thick, stainless steel sheet which was soldered to the face of
the copper block opposite that drilled for resistors. This stainless steel
sheet extended radially 3.1 mm beyond the copper cylinder where it was
soldered to a hollow stainless steel cylinder which was used as a vacuum
jacket. This jacket provided thermal insulation for the heating unit except
at the heat transfer surface.

The heater was mounted on a 0.95 cm diameter stainless steel thin-
walled tube, used as the vacuum line for the heater jacket; a good insulating
vacuum was maintained at all times. The tube was also used as a conduit

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Figure 11. Heat Transfer Unit

The figure contains the following labels:

Type KP vs Au (0.07 at. % Fe) Thermocouples

Heater Surface S.S. Sheet

Heater Resistors

Vacuum

Cold Hermetic Seal

Thermocouple Temperature Reference Block

Heater Copper Block

Heater Power and Voltage Leads

To Vacuum Pump
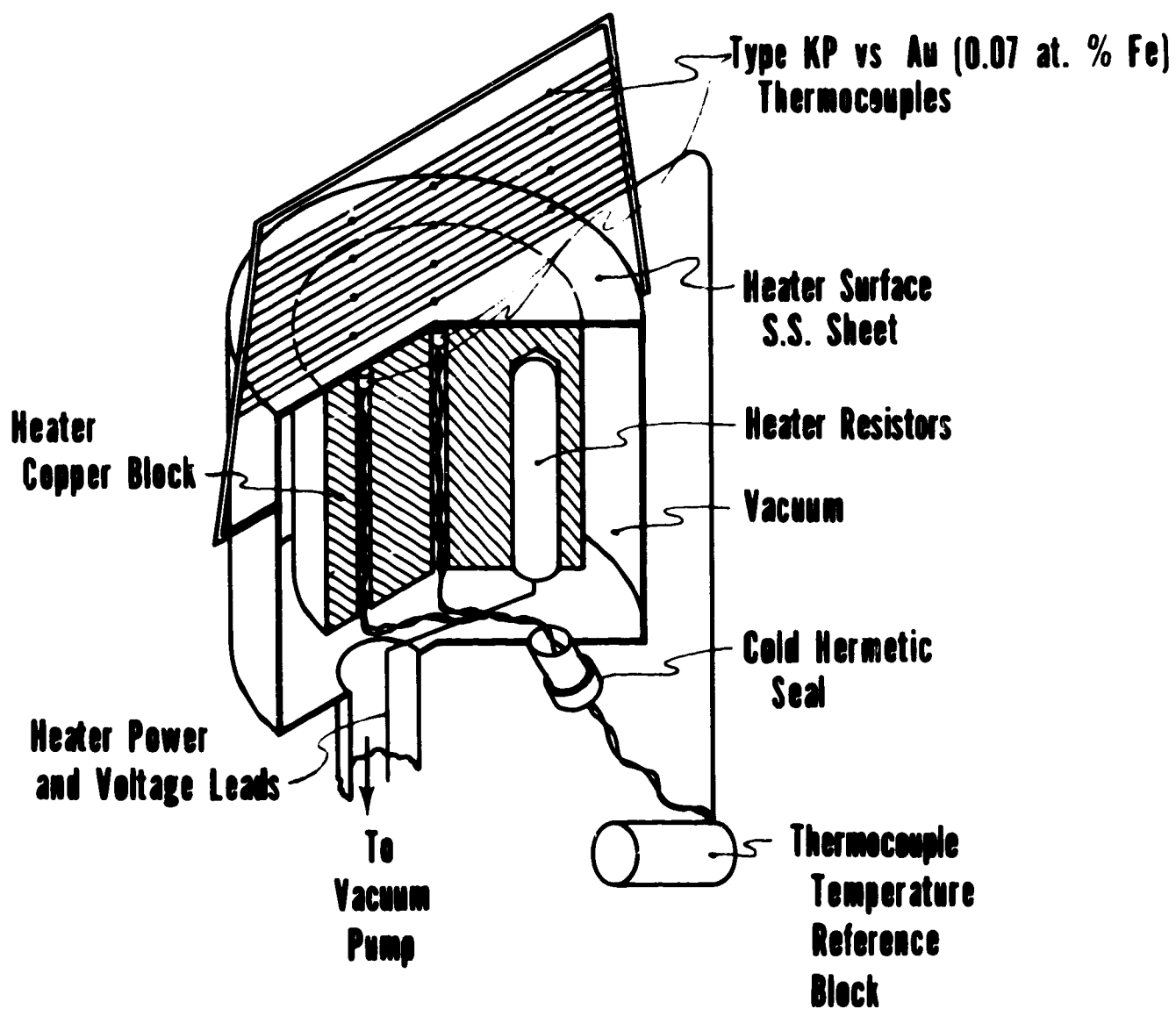
for the power leads and the voltage measurement leads. These leads were contained in the tube for about 30 cm and were, therefore, in a vacuum environment within the experimental vessel. At the top of the experimental vessel, the leads passed through a hermetic seal and were then enclosed in a second tube for about one meter length. This tube was filled with helium gas and was coiled in the liquid hydrogen bath dewar so that it remained submerged in liquid hydrogen. Submerging the assembly in the temperature controlled hydrogen bath intercepted the heat transferred down the wires before entering the experimental vessel and the heater unit.

3.2 Instrumentation

At 0.25 mm below the heat transfer surface, two thermocouples were embedded in a low melting temperature metallic eutectic. One thermocouple was located at the center of the heated surface; the other was at a radius of 0.96 cm and was centered between two of the resistance heaters. An array of twelve thermocouples extended outward from the external heated surface, spaced at approximately 1.1 mm intervals. Four of the thermocouples were located at the center line, four at about one half of the heater radius, and the other four at the heater edge. The thermocouple array is shown in the heater schematic in figure 11.

A second thermocouple array was initially mounted at the liquid-gas interface in the test vessel. The intent was to measure thermal stratification of the liquid during the tests. However, the unit interferred with slush preparation and no stratification developed in three of the four types of tests. In the case where stratification did develop, the data were meaningless since stratification was dependent on the frequency of slush preparation and this was a nearly random occurrence depending on many other variables. The thermocouples were all Type KP vs. gold (0.07 atomic percent iron).

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All thermocouples in the array and in the heater were referenced to a thermocouple located at the bottom of the test vessel. In this location the absolute temperature was known for each test. During normal-boiling-point testing, the dewar pressure was maintained at one atmosphere so the bulk liquid remained in equilibrium with the pressure and was at normal-boiling-point temperature. Since the reference thermocouple was below the heater, it remained in normal-boiling-point liquid. During triple-point liquid tests, the dewar was held at triple-point pressure and bulk liquid was at triple-point temperature. The bottom location of the reference thermocouple resulted in an increase in equilibrium temperature of less than 0. 01 K at one atmosphere, and 0. 05 K at triple point (due to liquid head). For tests using slush hydrogen, the reference thermocouple was always surrounded by slush hydrogen, so it was at triple-point temperature regardless of dewar pressure. Thermocouple signals were amplified 1000 times and recorded automatically on magnetic tape with a data acquisition system.

The power to the heater was provided by a d-c power supply which maintained voltage constant within ± 0. 2 percent. The voltage at the heater and the voltage drop across a precision resistor in series with the heater were measured to calculate the power supplied to the heater. Pressure was measured with an absolute pressure mercury manometer and was maintained constant within ± 135 N/m^2 using the barostat.

4. 0 Test Procedure

Four types of tests were conducted using three orientations of the heater surface. The four test types were: 1) heat transfer at one atmosphere pressure in liquid at normal-boiling-point temperature, 2) heat transfer at triple-point pressure in liquid hydrogen, 3) heat transfer at triple-point pressure in settled slush hydrogen (estimated solid

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fraction of 0.45), and 4) heat transfer in settled slush hydrogen at one
atmosphere pressure using helium as the pressurizing gas. The three
orientations of the surface were horizontal facing up, vertical, and
horizontal facing down. Test procedure did not vary for the three
orientations of the heater.

The test procedure for normal-boiling-point liquid started by
bringing all of the liquid to equilibrium at one atmosphere pressure.
The liquid was mixed until bubbles formed at the lower mixer blade
and did not collapse in route to the surface. At this time, all of the
thermocouples were read to establish a zero point offset. Power was
then supplied to the heater in increasing increments. The thermo-
couple data and the power were recorded while the pressure in the ves-
sel was maintained constant. Heat was increased from 0.002 W/cm^2
to the point where the boiling regime changed from nucleate to film
(burnout). The heating rate was increased by increments to the maxi-
mum, then decreased to determine hysteresis effects.

The procedure for tests of triple-point liquid was to pump the
dewar to near triple-point pressure and maintain this pressure without
forming solid. During the remainder of the test, triple-point-pressure
was maintained; otherwise, the test was the same as for normal-boiling-
point liquid.

The procedure for heat transfer to slush at triple-point pressure
was to prepare slush in the experimental dewar using the freeze-thaw
method; the dewar was filled with settled slush. Triple-point pressure
was then maintained with the barostat. Heat was increased and decreased
as for the liquid tests, but burnout was not defined, as the solid in the
slush could not be maintained long enough to determine the actual burn-
out heat rate with any certainty. Therefore, due to this apparatus

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limitation, the maximum heat rate used in the slush was arbitrarily selected as that used in the liquid.

For the slush pressurized to one atmosphere pressure, the procedure was similar, except that after slush preparation and prior to adding heat, the pressure was raised to one atmosphere by introducing cold helium gas. The pressure was maintained during the test. This test was always run last in the series so that liquid used in the other tests was not saturated with helium gas.

For both slush tests, the slush had to be replenished frequently, thus interrupting the increasing or decreasing heat rate. To maintain consistency after slush preparation, the heat rate was always increased from a lesser value for increasing heat flux tests and decreased from an arbitrary larger value for decreasing heat flux tests. During the higher heat flux tests, slush had to be prepared prior to each test point to assure adequate slush depth over the heater during the period required to take data.

5.0 Test Results and Discussion

At least two separate runs were made for each type of test and heater position to check for repeatability of tests and consistency of data. The temperature difference between the two thermocouples in the heater was monitored to assure that the heater surface was at a uniform temperature.

All of the test data were reduced to the parameters of power per unit of area versus the temperature difference between the bulk liquid and the heater surface. The power per unit of area was calculated from the power supplied to the heater and the diameter of the heater block. A correction was made for the heat loss through the stainless steel fin formed by the extension of the sheet from the copper block to

the vacuum jacket. A correction was also made to the temperature difference for the temperature drop from the copper block to the stainless steel surface exposed to the liquid. Both corrections were relatively small. Curves fit to the data for each type of test and test configuration are shown in figures 12 through 14. Actual test data points are shown for some of the test conditions to illustrate typical data scatter.

On the graph for the heater facing up position in figure 12, the curve of Coeling and Merte is also given for a pressure of 117 kN/m^2 (878 torr). The agreement is good. Coeling and Merte did not show a hysteresis effect for decreasing heat flux for a polished surface; their data for a rough surface did indicate a hysteresis similar to that shown for normal boiling liquid. No hysteresis was found in the other three types of tests for this heater position.

The point where the curves make a sharp break coincides with the time that vapor bubbles were first observed at the heater surface, except for the slush pressurized to one atmosphere. In this case, vapor bubbles were not visible; however, they may have formed and collapsed after leaving the surface and not have been observed, since visibility in slush is poor. Vapor sites were apparent during the decreasing heat flux portion of the normal boiling liquid tests. Three sites were still apparent at the lowest point. In the triple-point liquid test, three vapor sites were still visible at a temperature difference of two degrees, and one vapor site existed at 0.8 degrees. These sites apparently were not enough to affect the heat transfer rate and therefore did not result in the hysteresis effect that developed in normal-boiling-point liquid. Data for triple-point liquid and slush at triple-point pressure were not significantly different in this heater position.
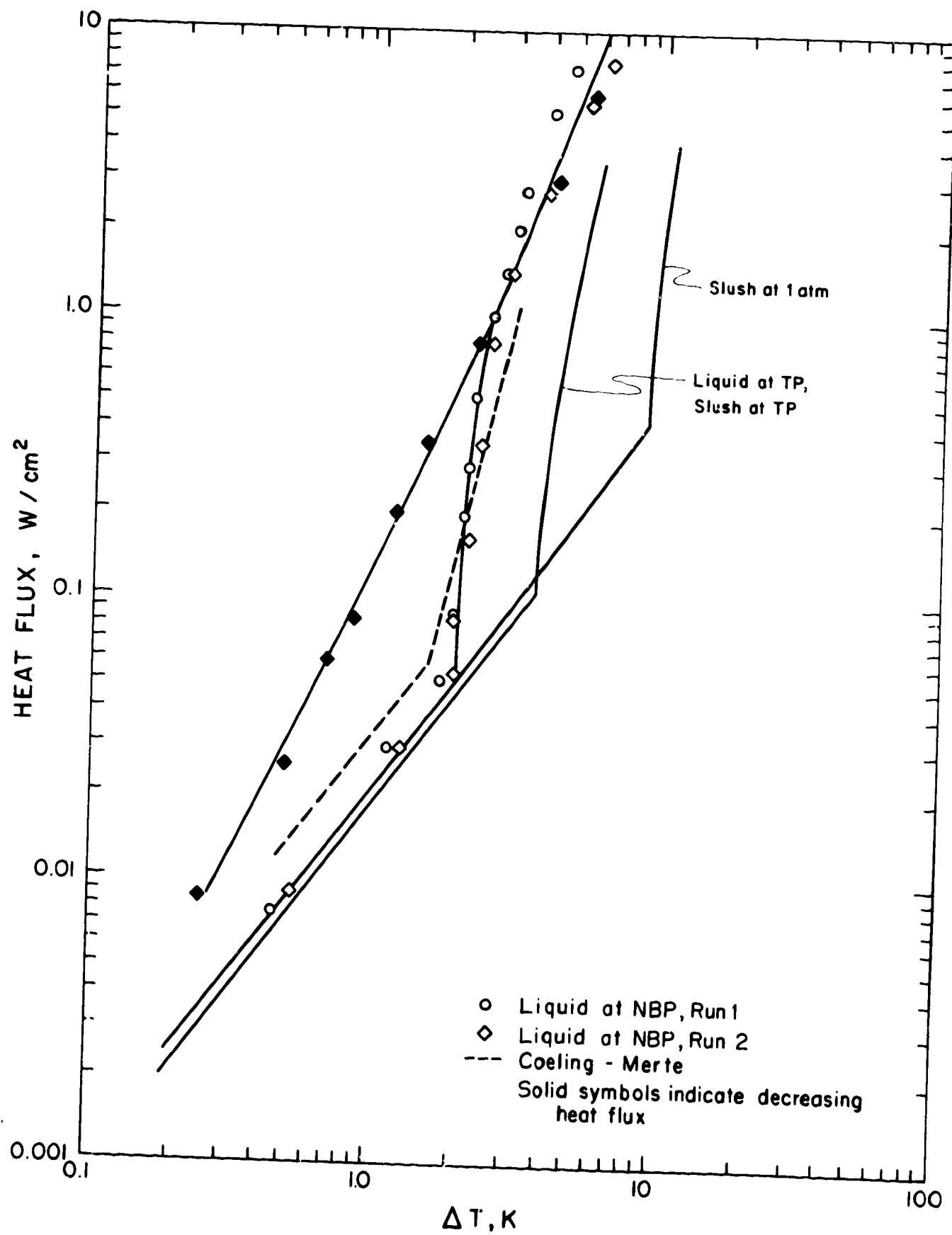
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Figure 12. Heat Transfer of Hydrogen, Horizontal Surface Facing Up.
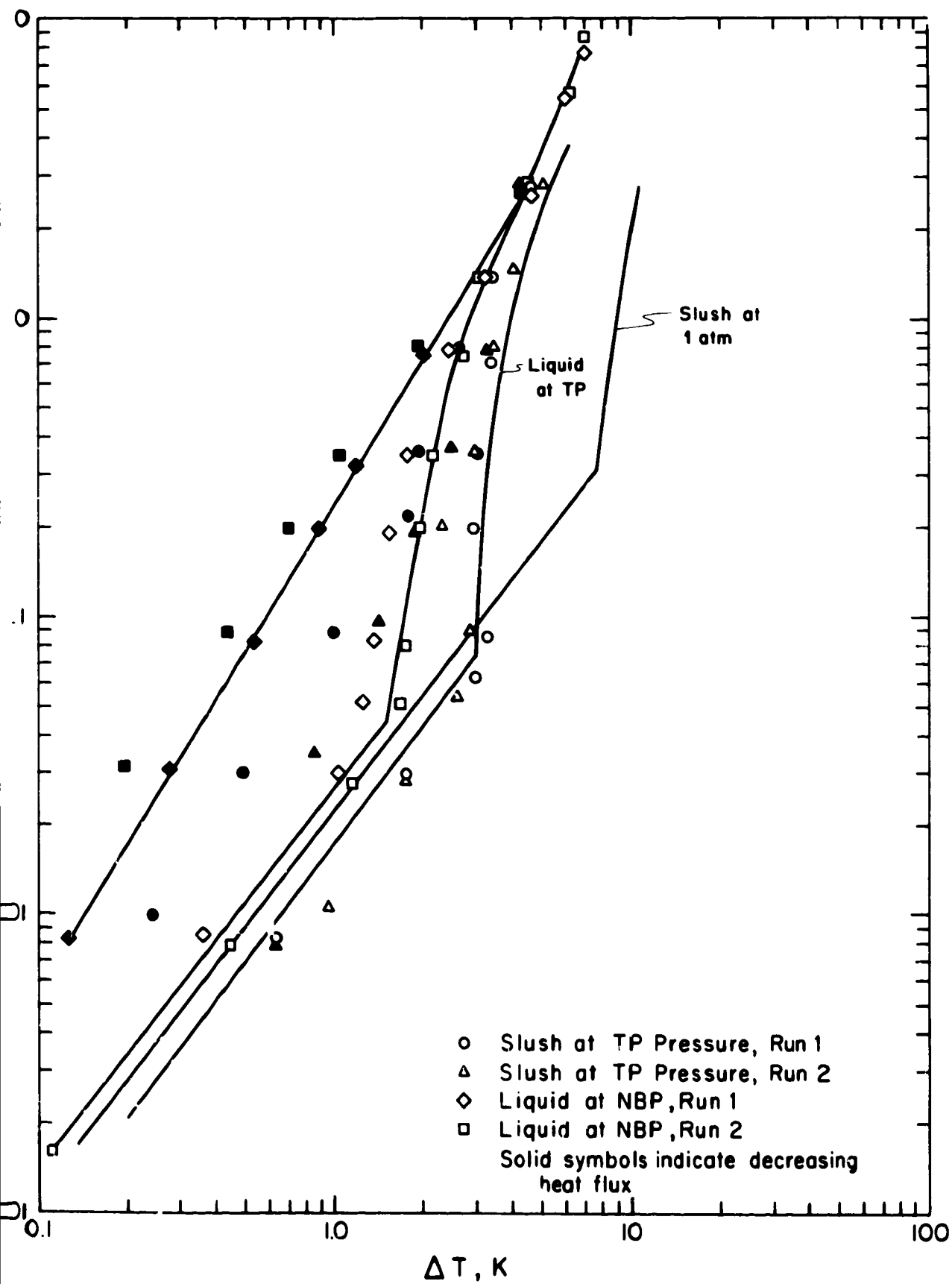
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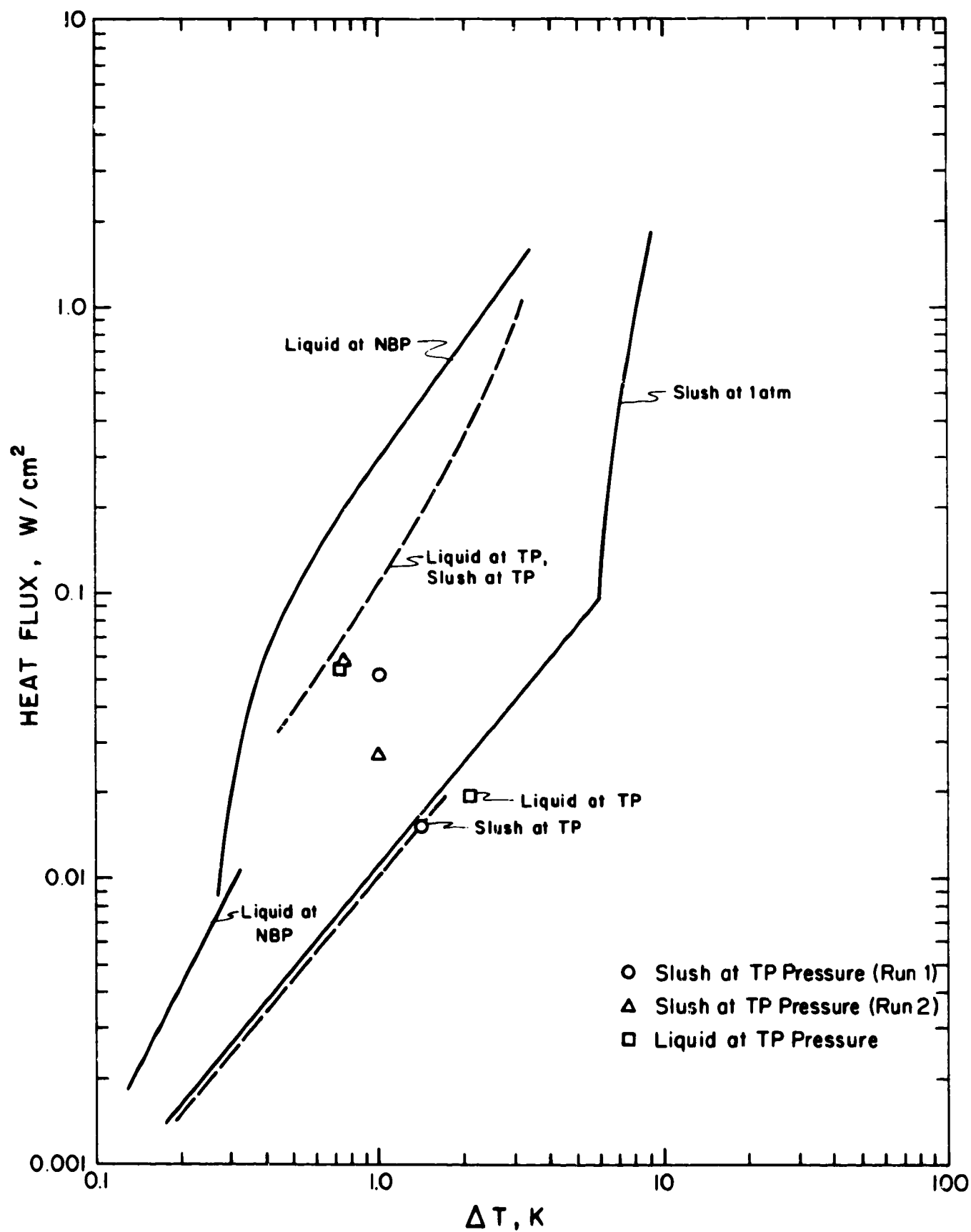
Figure 13. Heat Transfer of Hydrogen, Vertical Surface.

Figure 14. Heat Transfer of Hydrogen, Horizontal Surface Facing Down.

When the boiling became vigorous in slush at triple-point pressure, mixing of the slush resulted. The mixing started at a heat flux of 0.5 W/cm^2, and became vigorous at 0.8 W/cm^2. Mixing resulted from the vigorous bubble action in the boiling liquid. Since no bubbles persisted in the pressurized slush case, no apparent mixing occurred.

A significant difference exists between the heat transfer data for the heater facing up and the heater vertical; hysteresis was present in both triple-point liquid and slush at triple-point pressure with the heater in the vertical position, and the hysteresis was absent with the heater facing up. The data for slush of one atmosphere pressure were nearly the same. Visual observation of the vapor formation with the heater vertical revealed that the vapor sites always formed at the top of the heated surface first and progressed down across the surface as the heat flux was increased. This characteristic resulted because the fluid at the lower part of the heater flowed across the heater face as it warmed and became less dense. Since this fluid was slightly hotter than the bulk fluid and was receiving more heat as it ascended, the boiling point was reached at the upper most point of the heater first. Therefore, heat transfer to slush on vertically oriented surfaces may depend on the geometry. Apparently the 2.54 cm diameter surface was small enough so that the mean heat transfer characteristics were nearly the same as for a horizontal surface facing up.

Another characteristic that is apparent in the data for the vertical surface and for one set of data for the horizontal surface facing up is the second break in the curve for normal boiling liquid. This break occurred at the time vapor sites covered the entire surface. In the vertical position, this occurrence was repeatable and the data were consistent. In the horizontal facing up position, the vapor site formation was not consistent. On several occasions, sites appeared all across the surface

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simultaneously and vapor bubbles were very small and appeared as a cloud (data shown as circles in figure 12). At other times the vapor sites produced large bubbles and increased in number gradually as heat flux was increased (data shown as diamonds). This difference in bubble formation probably accounts for some difference in heat transfer rate although no large differences were observed.

The heat transfer characteristics in the heater facing down position were significantly different than for the other two positions. As shown in figure 14, the heat transfer to liquid at normal boiling temperatures is much larger at lower temperature differences. Vapor sites formed at the lowest heating rates, and a single large bubble grew until it escaped over the edge of the heater unit. Vapor sites did not form at the lower heating rates in liquid at triple point and slush at triple-point pressure. The large discontinuity in these curves coincided with the first visible vapor formation. The discontinuity in the data occurred because bubbles formed and covered the surface before escaping instead of escaping from vapor sites as in the other heater orientations. The hysteresis effects in this heater position were not as marked, probably because the entire surface was covered with a single vapor bubble as soon as boiling occurred. This same condition existed during decreasing heat flux.

The characteristics of pressurized slush for the facing down heater position were similar to characteristics of other orientations, but the heat flux was less for the same temperature difference. However, the break in the curve occurs at a lower temperature difference, and the temperature difference for a given heat flux is less from this point on.

The heater transfer in the free convection regime was examined further by comparing it to heat transfer in other fluids. The comparison

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was made using the dimensionless parameters of Grashof, Prandtl, and Nusselt numbers with the diameter of the heated surface used for the characteristic dimension. The data were compared to data and to equations given in Jakob [1949] for heat transfer to cylinders, vertical planes, spheres and a block in the fluids, air, water, alcohol, and oil. The results are given in figures 15, 16, and 17. The curve by Jakob is a curve fit to the data presented in Jakob for air, water, alcohol, and oil.

The data for normal boiling temperature liquid in the heater facing up orientation fit the equations with fair agreement. The data for triple-point liquid, triple-point slush, and slush at one atmosphere pressure in the horizontal facing up orientation are consistently displaced with the Nusselt number too large. The larger Nusselt number for horizontal facing up surfaces is suggested by Jakob [1949]. Values given by Jakob range from 28 to 100 percent larger than for vertical surfaces and for the geometric shapes represented by the equations. The triple-point liquid and slush data fit the curve quite well for the vertical orientation. In the horizontal facing down position, none of the data fit the curve or the equations. The data for normal-boiling-point temperature liquid were not expected to fit the correlations because vapor was forming during all of the data points; therefore, the fluid was not in a free convection state. For horizontal facing down surfaces, Jakob suggests Nusselt numbers as small as half of those for horizontal surfaces facing up. Nusselt numbers for the data shown in figure 17 are approximately one half of those for the facing up position shown in figure 15.

Heat transfer in the nucleate boiling regime was also compared to heat transfer in other fluids. The comparison was made using several different equations for predicting boiling heat transfer to water. These equations are given in Kutateladze [1952, 1963]. The comparison with heater facing up data is given in figure 18. A similar comparison by
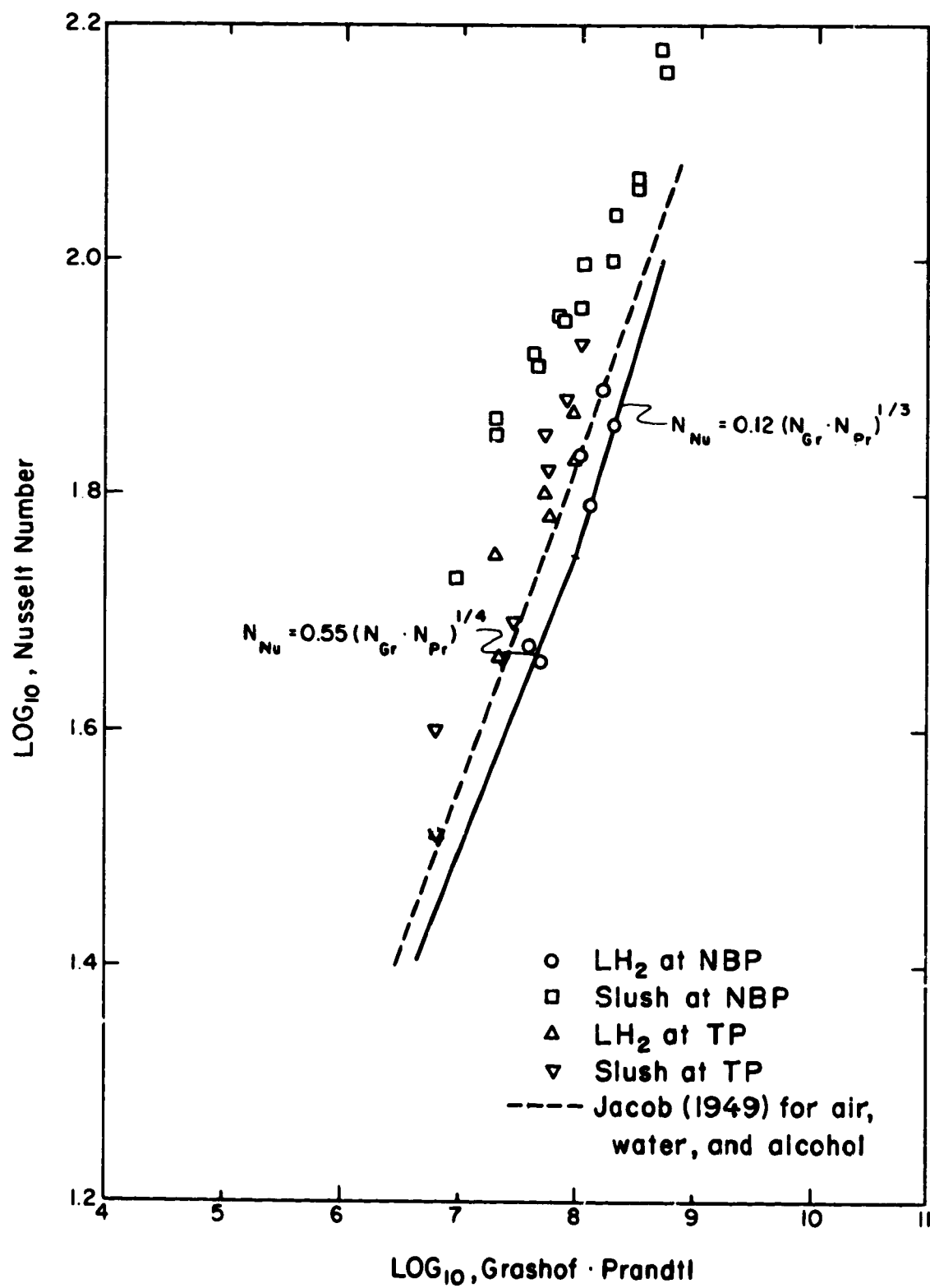
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Figure 15. Natural Convection Heat Transfer to Hydrogen, Horizontal Surface Facing Up

The figure contains the following labeled elements:

- Y-axis: LOG_{10}, Nusselt Number, ranging from 1.2 to 2.2
- X-axis: LOG_{10}, Grashof · Prandtl, ranging from 4 to 11
- $N_{Nu} = 0.12 (N_{Gr} \cdot N_{Pr})^{1/3}$
- $N_{Nu} = 0.55 (N_{Gr} \cdot N_{Pr})^{1/4}$

Legend:
- ○ LH_2 at NBP
- □ Slush at NBP
- △ LH_2 at TP
- ▽ Slush at TP
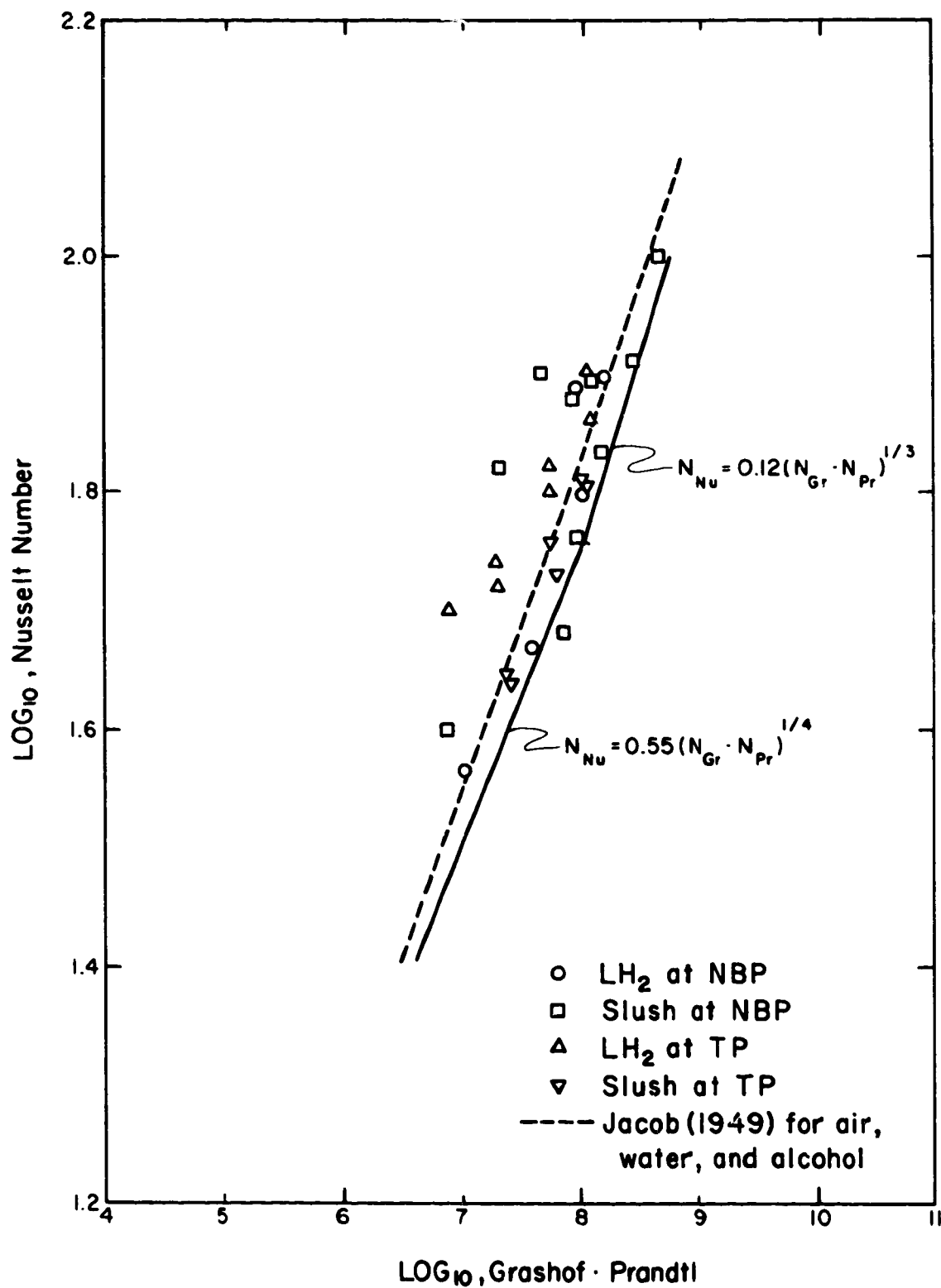- ---- Jacob (1949) for air, water, and alcohol

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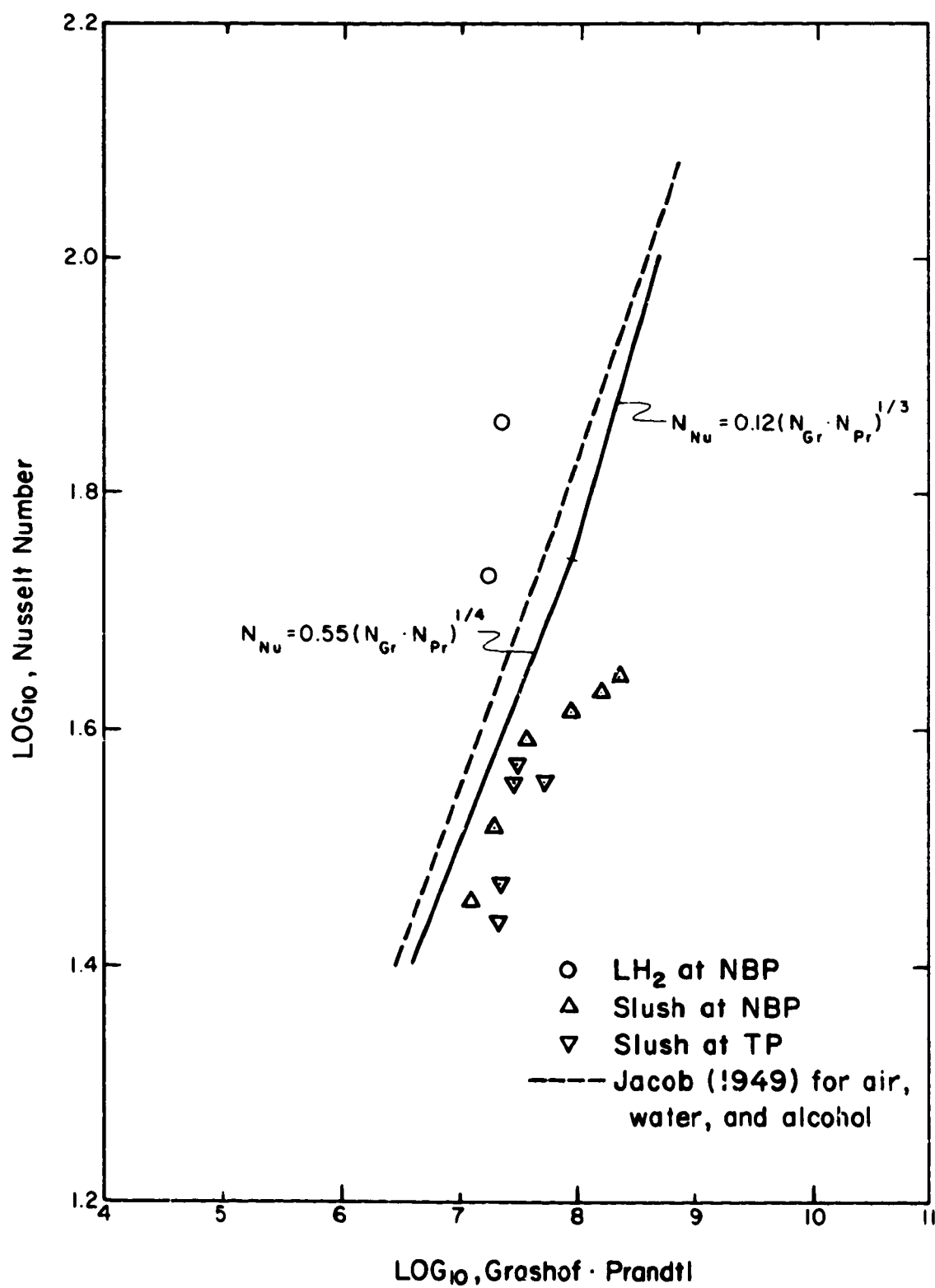Figure 16. Natural Convection Heat Transfer to Hydrogen, Vertical Surface

Figure 17. Natural Convection Heat Transfer to Hydrogen, Horizontal Surface Facing Down.

Brentari, et al., [1965] was made for hydrogen heat transfer data. The curves for predicted values given by Brentari are calculated using liquid thermal conductivity values from Scott [1964], while the thermal conductivity data used for the curves shown in figure 18 were from Roder and Diller [1970]. The significant difference in these thermal conductivity data account for the small difference between the curves given by Brentari and those shown in figure 18 for the identical equation.

The correlations of Kutateladze [1963] and Kichigin (from Kutateladze, 1963] agree with the normal boiling liquid data; however, these correlation do not fit the triple-point liquid data. All of the correlating equations predict too much effect on the temperature difference for the pressures of one atmosphere and triple point. Figure 19 shows the nucleate boiling regions covered by the three heater orientations and the prediction of Kutateladze [1963]. In general, the data do not show as much change in the temperature difference for a given heat flux as the equation predicts. No correlations for predicting the subcooled region were found, except for peak heat flux values. Since the only subcooled experimental data obtained were for slush, the peak heat flux was not applicable.

The only tests in which more than one degree temperature gradient developed in the hydrogen (at the first thermocouple in the array over the heater) was in slush pressurized to one atmosphere. In these tests, a difference was present as far out as the third thermocouple, or at a distance of approximately 3.3 mm. For the heater facing up position, at a heat flux of 4.5 W/cm^2, the temperature of the first thermocouple (1.1 mm from the surface) was 19.5 K, or 5.7 K above the bulk tempera- ture. The temperature of the second thermocouple at 2.2 mm was 17.7 K, and that of the third thermocouple was 13.9 K. In the other two positions, the temperature differences were higher at the maximum
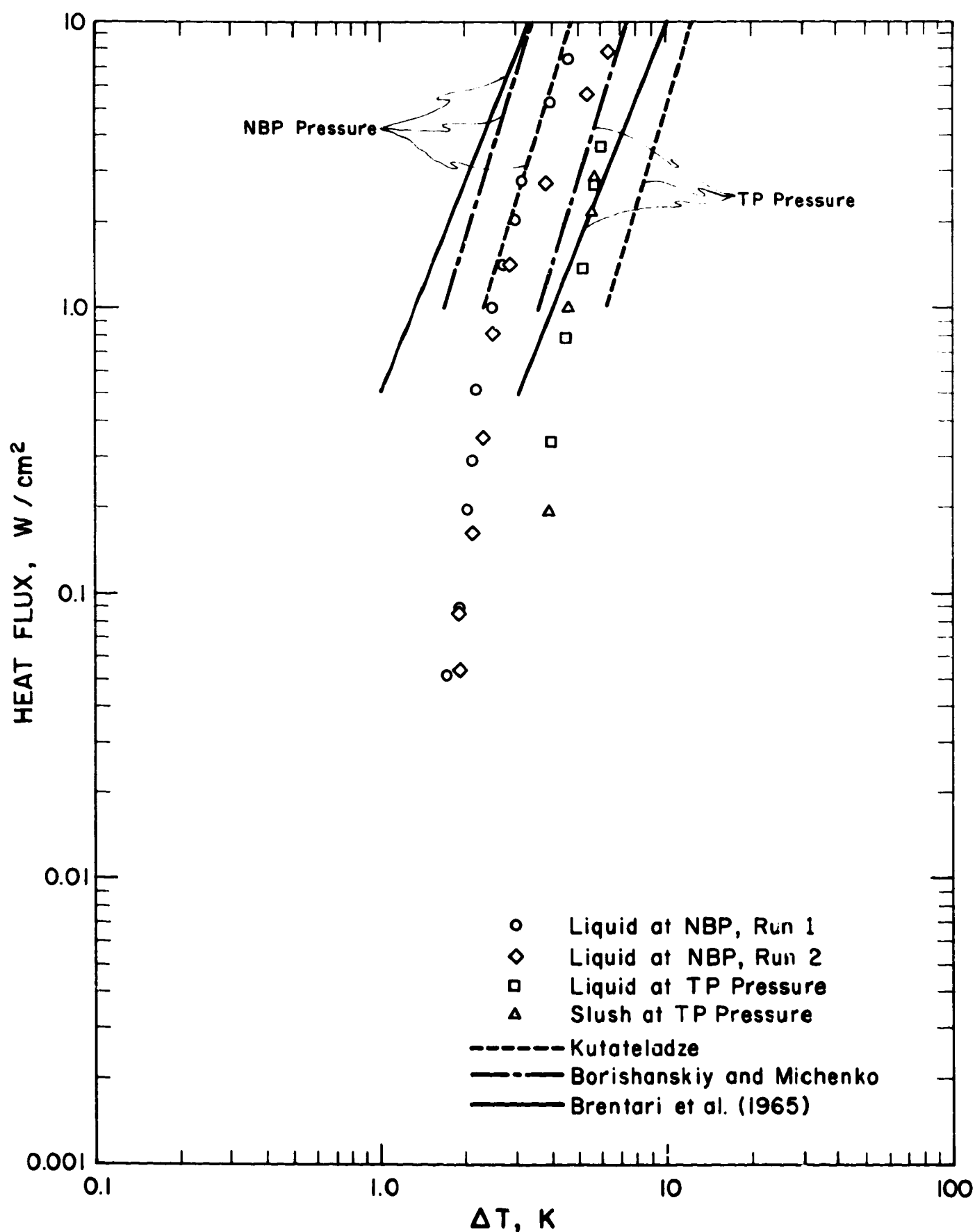
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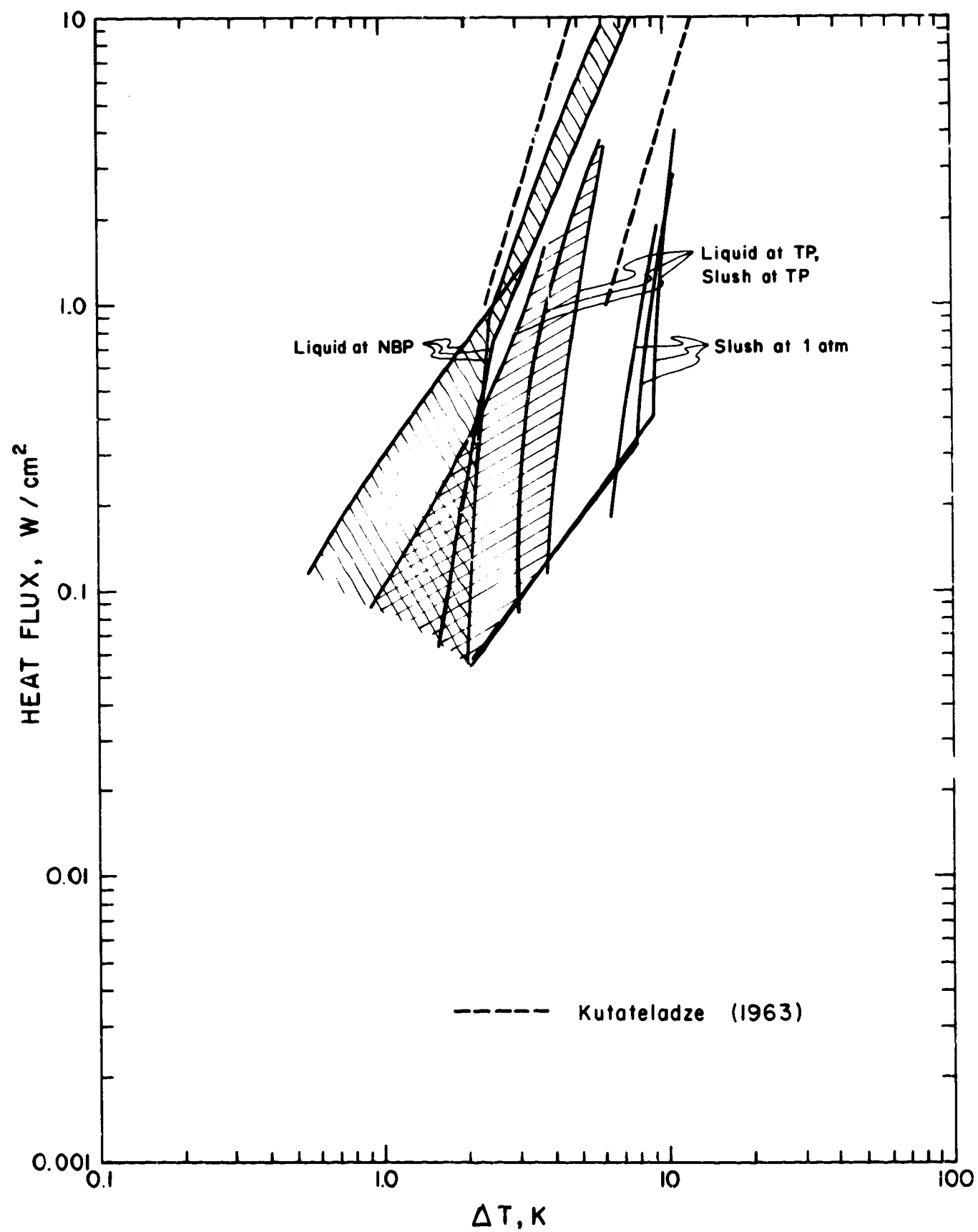Figure 18.   Correlations for Nucleate Boiling Heat Transfer to Hydrogen

Figure 19. Nucleate Boiling Heat Transfer to Hydrogen

heating rate, even though the maximum rate was lower than the heater facing up position. At 2. 9 W/cm^2, the maximum for the vertical heater position, the first thermocouple indicated 19. 5 K; and at 1. 9 W/cm^2, the maximum for the facing down heater position, the temperature was also 19. 5 K. The third thermocouple reached its maximum temperature of 14. 5 K during the facing down heater position. The fourth thermocouple never did indicate a significant difference from the bulk temperature.

6. 0 Conclusions

The natural convection heat transfer to liquid hydrogen at normal-boiling-point temperature can be predicted quite accurately for horizontal surfaces facing up and on vertical surfaces. Heat transfer in this regime can be predicted by the classical methods using Grashof, Prandtl, and Nusselts numbers. Since no truly natural convection data were obtained for the surface facing down, no comparison is available. The natural convection heat transfer for triple-point liquid, triple-point slush, and slush at one atmosphere are also predictable from the classical methods using Grashof, Prandtl, and Nusselt numbers. The correlating equations and the correction for surface orientation given by Jakob [1949] can be used with good agreement to predict the heat transfer for these three liquid and slush hydrogen conditions.

Heat transfer rates are very nearly the same for liquid and slush at triple-point temperature and pressure. This similarity is true over the entire range of natural convection and nucleate boiling, and for all heater positions.

At one atmosphere pressure, the heat transfer to slush remains in the convective mode until the surface temperature is one and one-half to three degrees above normal-boiling-point temperatures except for

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the facing down position. In the facing down heater position, the boiling mode starts when the surface temperature is about one-half degree below normal-boiling-point temperature. If the temperature difference above the equilibrium temperature is the basis for comparison, the required difference for the start of the boiling mode is nearly the same as for normal-boiling-point liquid; however, the heat flux in pressurized slush is significantly greater at the start of nucleate boiling. This is the case for all heater orientations.

Prediction of heat transfer rates in the nucleate boiling regime was not satisfactory with existing equations. Kutateladze's [1963] equation predicts the rates for the surface facing up in normal-boiling-point liquid if nucleation sites cover the surface. The equation does not predict rates in triple-point liquid. Since boiling heat transfer is highly sensitive to surface finish and textures, predicting nucleate boiling heat transfer without some surface condition correlation probably is not very accurate.

Several existing correlations do predict heat transfer rates on a smooth surface for normal-boiling-point liquid hydrogen in the nucleate boiling regime, but these same correlations do not predict heat transfer rates for triple-point liquid hydrogen. Those correlations that predict the boiling heat transfer rates to triple-point liquid with fair accuracy do not predict the rates to normal boiling liquid.

No correlations were found to predict heat transfer to liquid and solid mixtures of the same material. Heat transfer to slush hydrogen can be approximated by using triple-point liquid values for slush at triple-point temperature. For slush at higher pressure, the convective heat transfer can be approximated using the classic method. Heat transfer continues in the convective regime until the surface temperature is two to three degrees above the equilibrium temperature for the pressure.

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For the nucleate boiling regime, the heat transfer in slush at pressures above triple-point cannot be predicted accurately. A crude approximation can be made by drawing a line at 80 degrees to the abscissa on a graph similar to those of figures 12, 13, and 14. The convective heat transfer curve would be constructed first and would end at a temperature difference corresponding to two degrees above equilibrium temperature for the desired operating pressure. The nucleate boiling curve would be drawn from this point at 80 degrees from the abscissa axis. This approximation is based on data for one atmosphere pressure; therefore, it should not be used for extrapolation much above this pressure.

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